

Application No. 10/076,976

25. The collection of nanoparticles of claim 18 wherein at least about 95 percent of the primary particles have ratios of the dimension along the major axis to the dimension along the minor axis less than about two.

26. The collection of nanoparticles of claim 18 wherein the particles comprise rutile titanium dioxide and anatase titanium dioxide.

27. The collection of nanoparticles of claim 18 wherein the particles are dispersed in an aqueous solution.

28. The collection of nanoparticles of claim 18 wherein the particles are dispersed in a non-aqueous solution.

29. The collection of nanoparticles of claim 18 wherein the nanoparticles have an average diameter from about 5 nm to about 100 nm.

D4 30. (Amended) The collection of nanoparticles of claim 18 wherein the nanoparticles have an average diameter from about 5 nm to about 25 nm.

#### REMARKS

Claims 18-28 are pending. By this Amendment, claims 18, 19 and 30 are amended. The specification was amended to update the reference to the parent patent application. The amendments of claims 18, 19 and 30 are supported by the specification, for example, at page 17, lines 11-16 and page 24, lines 30-32. No new matter is introduced by the amendments of the claims or the specification.

Application No. 10/076,976

All of the pending claims stand rejected. Applicants respectfully request reconsideration of the rejections based on the following remarks.

Rejections Over Wiederhöft et al.

The Examiner rejected claims 18-30 under 35 U.S.C. § 102(b) as being unpatentable over U.S. Patent 5,840,111 to Wiederhöft et al. (the Wiederhöft patent). In the Amendment of October 10, 2002, Applicants presented evidence that the process of the Wiederhöft patent only related to the production of anatase titanium oxide and not to rutile titanium oxide. In response to Applicants' arguments, the Examiner asserts that "[t]o accept applicant's assertion is equivalent to invalidating the Wiederhöft reference based on a false disclosure. It is not within the Examiner's authority to do so." However, the Examiner's statements do not provide a possibility for advancing prosecution of the case and are contrary to well established legal principles. Applicants respectfully request reconsideration of the rejection based on the following comments.

In response to the prior Office Action of July 3, 2002, Applicants presented evidence that rebutted assertions that the Wiederhöft patent taught rutile titanium oxide with the claimed characteristics.

First, the claims of the Wiederhöft patent do not explicitly recite rutile titanium oxide. The Examiner must either refute Applicants' evidence or allow the claims. If Applicants' evidence is not convincing, the Examiner should point to any shortcomings of Applicants' evidence such that any concerns can be addressed. In particular, the Examiner must present either references that refute Applicants' evidence or present an affidavit under 37 C.R.F. 1.104(d)(2).

If the Examiner does not have authority to act on the case, presumably someone in the Office has appropriate authority to advance the case, and Applicants respectfully request

Application No. 10/076,976

directing the file to a person with the required authority. Since the Patent Office has the burden of establishing unpatentability, the case must be allowed if the Office does not refute Applicants' evidence.

With respect to Applicants' position, Applicants are not asserting a lack of enablement for the Wiederhöft patent. While Applicants' take the position that the Wiederhöft patent is ambiguous with respect to rutile titanium oxide since there is no connection between the crystal structure and the particle properties. Under MPEP 2121.02 a reference is only presumed operable until an applicant presents evidence rebutting the presumption of operability. Applicants Based on evidence presented by Applicants, the Wiederhöft patent does not prima facie anticipate Applicants' claimed invention, even assuming *arguendo* that Applicants are challenging operability, which they are not.

Due to the evidence presented by Applicants', the Wiederhöft patent does not render Applicants' claimed invention prima facie anticipated. Applicants respectfully request withdrawal of the rejection of claims 18-30 under 35 U.S.C. § 102(b) as being unpatentable over the Wiederhöft patent.

Rejection Over Montino et al. Or Colombo et al.

The Examiner rejected claims 18-21 and 23-28 under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over either U.S. Patent 4,803,064 to Montino et al. (the Montino patent) or U.S. Patent 3,661,522 to Colombo et al. (the Colombo patent). The Examiner cited the reference for disclosing rutile titanium dioxide with a uniform particle size distribution. The Examiner asserted that the particles would inherently have the claimed particle size distribution. The Examiner further asserted that it would have been obvious for one having ordinary skill in the art to select particles within the claimed range by simple mechanical process such as sieving or filtering. To advance prosecution of the case, Applicants have amended the claims to more particularly point out their claimed invention. In

Application No. 10/076,976

view of these amendments, the Montino patent and the Colombo patent do not render the claims prima facie unpatentable. Applicants respectfully request reconsideration of the rejection based on the following comments.

With respect to the Montino patent, Applicants assert that there is no basis for assuming that the materials have the particles size distribution of Applicants' claims based on general statement about particle uniformity. However, to advance prosecution of the application, Applicants have amended claim 18 to more precisely indicate Applicants' claimed invention. The Montino patent does not disclose or suggest rutile titanium dioxide particles with an average particle size of 150 nm or less. See, for example, column 5, lines 45-47. Therefore, the Montino patent does not render Applicants' claimed invention prima facie unpatentable.

With respect to the Colombo patent, the distribution of particle sizes is described as following a normal distribution. See column 2, lines 31-53. Based on the results in the examples, this distribution does not lead to Applicants' claimed distribution, which is not a normal distribution. Nevertheless, Applicants have amended claim 18 to advance prosecution. The Colombo patent does not teach or suggest rutile titanium dioxide with an average particle size no more than about 150 nm. See column 2, lines 63-65. Therefore, the Colombo patent does not render Applicants' claimed invention prima facie unpatentable.

Since neither the Montino patent nor the Colombo patent teach Applicants' claimed particle size and particle size distribution, the references alone or combined do not render Applicants' claimed invention prima facie obvious. Applicants respectfully request withdrawal of the rejection of claims 18-21 and 23-28 under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over either the Montino patent or the Colombo patent.

Application No. 10/076,976

Rejection Over Bruno

The Examiner rejected claims 18-30 under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over U.S. Patent 5,776,239 to Bruno (the Bruno patent). The Examiner asserts that the Bruno patent discloses rutile titanium dioxide particles with the claimed average particle size and a uniform distribution. The Examiner further asserts that the Bruno patent inherently has the claimed particle size distribution. In the alternative, the Examiner asserts that a person of ordinary skill in the art could select the claimed particle size range by simple mechanical processes such as sieving or filtering. To advance prosecution of the case, Applicants have amended claim 18 to more particularly point out their claimed invention. In view of the amendments, the Bruno patent clearly does not render Applicants' invention prima facie unpatentable. Applicants respectfully request reconsideration of the rejection based on the following comments.

First, the particle sizes referred to by the Examiner are not particle sizes. By the Bruno patent's own statements, these are crystallite sizes, i.e., the sizes of the crystals within polycrystalline particles/agglomerates. See column 2, lines 27-29 and column 4, lines 59-61. The agglomerates have an average particle size on the order of 300 nm. See column 4, lines 61-62, column 9, lines 35-40 (0.17 microns or 170 nm), and column 10, lines 46-48 (0.28 microns or 280 nm). Thus, the Bruno patent does not teach or suggest titanium dioxide particles with an average particle size of no more than 150 nm.

Furthermore, "[w]hen the PTO seeks to rely upon chemical theory, in establishing a prima facie case of obviousness, it must provide evidentiary support for the existence and meaning of that theory. In re Mills, 47 CCPA 1185, 1191, 281 F.2d 218, 223-224, 126 USPQ 513, 517 (1960)." In re Grose, 201 USPQ 57, 63 (CCPA 1979)(emphasis added). The Examiner has stated without support the proposition that inorganic nanoparticles can be filtered or sieved to form highly uniform distributions of particles. Applicants enclose evidence that state of

Application No. 10/076,976

the art filtration technology, subsequent to the priority date of the present application, from industry leader Millipore Corp. would not be capable of performing the described separation. Furthermore, Applicants' have additional evidence refuting the possibility of filtering for nanoscale particles to obtain more uniform particles. However, Applicants' believe that the Examiner has fallen short of meeting the PTO's burden of providing adequate support for the asserted proposition relating to particle separation by unsupported aspirational statements. Thus, Applicants do not need to present any further evidence. Applicants respectfully request appropriate support for the filtering suggestion or a withdrawal of the rejection. If the Examiner is relying on personal knowledge, such support can take the form of an Affidavit under 37 C.F.R. 1.104(d)(2), such that Applicants can provide suitable counter evidence.

Since the Bruno patent does not disclose the claimed average particle size nor has the Examiner supported assertions regarding obtaining the claimed particle size distribution, the Bruno patent does not render Applicants' claimed invention prima facie unpatentable. Applicants respectfully request withdrawal of the rejection of claims 18-30 under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over the Bruno patent.

Rejections Over Siegal et al., Hansenne et al., Okuda et al. or Pratsinis et al.

The Examiner rejected claims 18-30 under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent 5,320,800 to Siegal et al. (the Siegal patent), U.S. Patent 5,772,987 to Hansenne et al. (the Hansenne patent), U.S. Patent 5,837,050 to Okuda et al. (the Okuda patent) or U.S. Patent 6,254,940 to Pratsinis et al. (the Pratsinis patent). The Examiner asserts that the cited references teach rutile titanium dioxide with the claimed average particle size. The Examiner notes that the references do not report a particle size distribution. However, the Examiner asserts that it would be obvious for one of ordinary skill in the art to select particles within the claimed range by simple mechanical processing such as sieving or filtering. The Examiner further asserts that Applicants have not disclosed that the specific particle size

Application No. 10/076,976

distribution serves any particular purpose. Applicants maintain that the Examiner has not asserted a case for prima facie obviousness. Applicants respectfully request reconsideration of the rejection based on the following comments.

Siegel Patent and Pratsinis Patent

The Siegel patent and the Pratsinis patent do not teach or suggest nanoparticles as claimed by Applicants. Specifically, with respect to the Siegel patent, the patent describes "nanocrystalline" materials and not nanoparticles. See, for example, the abstract. Thus, the dimensions described are grain sizes and not particle sizes. See, for example, column 5, lines 49-56. The Siegel patent does not describe rutile titanium dioxide particles with an average particle size as disclosed and claimed by Applicants. Therefore, the Siegel patent clearly does not render Applicants' claimed invention obvious.

With respect to the Pratsinis patent, the patent described primary particle size, but these particles are described as being agglomerated. At column 18, line 65 to column 19, lines 12, the Pratsinis patent describes the agglomeration of the primary particles to form agglomerates, i.e., hard fused particles. In particular, an increase in agglomeration is correlated with a decrease in primary particle size. The primary particle size seems to be only evaluated by the surface area, which does not directly correlate at all with real particle size since porous large particles can have a very high surface area. Thus, Applicants assert that the Pratsinis patent does not teach or suggest particles with an average particle size within the range of Applicants' claims. Since the Pratsinis patent does not teach particles in the claimed average particle size range, the Pratsinis patent does not render Applicants' claimed invention obvious.

Hansenne Patent And Okuda Patent

The Examiner asserts that the uniformity of the materials described the Hansenne patent and the Okuda patent can be purified to form the claimed materials. However, the Examiner has not described the motivation to modify the materials. Even if such motivation for

Application No. 10/076,976

highly uniform particles is presented, Applicants have already refuted the Examiner's assertions about the ability in the art to perform such purification, see above.

The motivation, or suggestion, to combine or modify references must be either explicitly or implicitly in the references or knowledge "generally available to one of ordinary skill in the art." See, MPEP § 2143.01. Furthermore, "[t]he test for an implicit showing is what the combined teachings, knowledge of one of ordinary skill in the art, and nature of the problem to be solved as a whole would have suggested to those of ordinary skill in the art." See, MPEP §2143.01 (quoting *In re Kotzab*, 55 USPQ2d 1313, 1317 (Fed. Cir. 2000)).

The Federal Circuit has provided considerable guidance on establishing obviousness of a claim based on a combination of references. "Our case law makes clear that the best defense against hindsight-based obviousness analysis is the rigorous application of the requirement of a teaching or motivation to combine the prior art references." *Ecolochem Inc. v. Southern Edison*, 56 USPQ2d 1065, 1073 (Fed. Cir. 2000). "Therefore, '[w]hen determining the patentability of a claimed invention which combines two known elements, the question is whether there is something in the prior art as a whole to suggest the desirability, and thus the obviousness, of making the combination.'" *Id.* (quoting *In re Beattie*, 24 USPQ2d 1040, 1042 (Fed. Cir. 1992)(quoting *Lindemann Maschinenfabrik GmbH v. American Hoist and Derrick Co.*, 221 USPQ 481, 488 (Fed. Cir. 1984))). "In order to prevent a hindsight-based obviousness analysis, we have clearly established that the relevant inquiry for determining the scope and content of the prior art is whether there is a reason, suggestion, or motivation in the prior art or elsewhere that would have led one of ordinary skill in the art to combine the references." *Ruiz v. A.B. Chance Co.*, 57 USPQ2d 1161, 1167 (Fed. Cir. 2000). **"The test is not whether one device can be an appropriate substitute for another."** *Id.* (emphasis added). In *Ruiz*, the Federal Circuit overturned a district court holding that "it would have been obvious to combine screw anchors and metal brackets, because the need for a bracket 'was apparent.'" *Id.*

Application No. 10/076,976

With respect to the Hansenne patent and the Okuda patent, the patents refer to the use of commercially available pigments. Also, it is not at all clear if these patents are referring to real particles or primary particles relating to crystallite size. However, there are other shortcomings with respect to the rejection. In particular, the Examiner has not pointed to any motivation within the Hansenne patent or the Okuda patent to suggest the desirability of forming highly uniform particle collections. Thus, prima facie obviousness is not established because the required motivation has not been described. Furthermore, the Examiner's unfounded assertions regarding filtering and sieving are refuted above. Therefore, the Examiner has fallen far short of establishing prima facie obviousness of the claimed invention.

If the Examiner fails to establish a prima facie case of obviousness, the obviousness rejection must be withdrawn as a matter of law. In re Ochiai, 37 USPQ at 1131 ("When the references cited by the examiner fail to establish a prima facie case of obviousness, the rejection is improper and will be overturned."). Since the Examiner has fallen short of establishing prima facie obviousness, Applicants have no need to supply other objective evidence of non-obviousness. However, the Examiner asserts that Applicants have not presented any problem solved by particles with the claimed particle size distribution.

While Applicants have no need to provide evidence of improved performance of their claimed materials, such evidence is supplied with this response. In particular, the nanoscale of the particles shifts the absorption spectrum of the particles from the visible to the ultraviolet. By having more uniform particle sizes, the spectrum further narrows with reduced visible absorption and an enhanced ultraviolet absorption. The ultraviolet (UV) and visible absorption properties of titanium oxide nanoparticles formed by laser pyrolysis are described in another pending U.S. case and in published PCT application WO 02/058928. An example from this application is enclosed with this Amendment. The improved absorption properties of substantially uniform titanium dioxide nanoparticles can be seen in the examples. The improved UV absorption has

Application No. 10/076,976

obvious application for ultraviolet absorbing materials and the decreased visible absorption has obvious advantages in for forming transparent materials such as windows or optical devices.

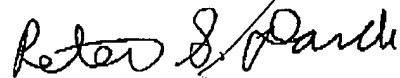
In summary, the Examiner has failed to establish prima facie obviousness of Applicants' claimed invention over any of the four cited references. In particular, several, if not all, of the references do **not** disclose particles within the claimed average particles size range. Furthermore, the modifications suggested by the Examiner are unsupported and unmotivated by the relevant references. Furthermore, Applicants have presented evidence of the improved optical performance of Applicants' claimed materials. Applicants respectfully request withdrawal of the rejection of claims 18-30 under 35 U.S.C. § 103(a) as being unpatentable over the Siggal patent, the Hansenne patent, the Okuda patent or the Pratsinis patent.

#### CONCLUSIONS

In view of the foregoing, it is submitted that this application is in condition for allowance. Favorable consideration and prompt allowance of the application are respectfully requested.

The Examiner is invited to telephone the undersigned if the Examiner believes it would be useful to advance prosecution.

Respectfully submitted,



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Application No. 10/076,976

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February 17, 2003

Date



Shari R. Thorndike

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Application No. 10/076,976

ATTACHMENT  
REDLINED AMENDMENTSpecification As Amended

In the Cross Reference To Related Applications, the paragraph has been amended as follows. This paragraph was added in the Preliminary Amendment of February 15, 2002.

This application is a continuation of copending and commonly assigned U.S. Patent Application Serial Number 09/123,255, now U.S. Patent 6,387,531 to Bi et al., entitled "Metal (Silicon) Oxide/Carbon Composite Particles," incorporated herein by reference.

Claims As Amended

Claims 18, 19 and 30 have been amended as follows:

18. (Twice Amended) A collection of nanoparticles, the nanoparticles comprising rutile titanium dioxide, wherein the particles have an average diameter no more than about 150 nm and effectively no particles have a diameter greater than about four times the average diameter.
19. (Amended) The collection of nanoparticles of claim 18 wherein the nanoparticles have an average diameter from about 5 nm to about [100]125 nm.
30. (Amended) The collection of nanoparticles of claim 18 wherein the nanoparticles have an average diameter from about 5 nm to about [500] 25 nm.

From U.S. 6,387,531

Part  
# 100Example - Titanium Oxide

The synthesis of titanium dioxide particles described in this example was performed by laser pyrolysis.

The titanium tetrachloride (Strem Chemical, Inc., Newburyport, MA) precursor vapor was carried into the reaction chamber by bubbling Ar gas through  $\text{TiCl}_4$  liquid in a container at room temperature.  $\text{C}_2\text{H}_4$  gas was used as a laser absorbing gas, and argon was used as an inert gas. The reaction gas mixture containing  $\text{TiCl}_4$ , Ar,  $\text{O}_2$  and  $\text{C}_2\text{H}_4$  was introduced into the reactant gas nozzle for injection into the reaction chamber. The reactant gas nozzle had an opening with dimensions as specified in the last row of Table 1. The production rate of titanium dioxide particles was typically about 4 g/hr. Additional parameters of the laser pyrolysis synthesis relating to the particles are specified in Table 1.

TABLE 1

Sample	1	2	3	4
Crystalline Phase	Anatase	Anatase & Rutile	Anatase	Anatase & Rutile
Crystal Structure	Tetra-gonal	Tetra-gonal	Tetra-gonal	Tetra-gonal
Pressure (Torr)	220	110	350	320
Argon - Win. (sccm)	700	700	700	700
Argon - Sld. (slm)	5.6	2.8	4.2	7.92
Ethylene (sccm)	603	603	603	1340
Carrier Gas (Ar) (sccm)	196	196	283	714
Oxygen (sccm)	252	382	300	550
Laser Output	520	520	520	450

(watts)				
Nozzle Size	5/8in X 1/8in	5/8in X 1/8in	5/8in x 1/8in	5/8in x 1/8in

sccm = standard cubic centimeters per minute

slm = standard liters per minute

Argon - Win. = argon flow through inlets 216, 218

Argon - Sld. = argon flow through annular channel 142

To evaluate the atomic arrangement, the samples were examined by x-ray diffraction using the Cu(K $\alpha$ ) radiation line on a Siemens D500 x-ray diffractometer. X-ray diffractograms for samples produced under the conditions specified in the four columns of Table 1 are shown in Figs. tio-1 to tio-4, respectively. Under the conditions specified in columns 1 and 3 of Table 1, the titanium dioxide components of the particles had a crystalline phase of anatase titanium dioxide. The diffractograms in Figs. tio-1, tio-3 and tio-4 have broad peaks at about 23° and at low scattering angles indicative of amorphous carbon.

In comparison, the titanium dioxide particles produced under the conditions shown in the second and fourth column of Table 1 had a crystal structure indicating mixed phases of anatase and rutile titanium dioxide. Under the conditions specified in the fourth column of Table 1 only a small portion of rutile titanium dioxide was formed. Furthermore, the diffractogram in Fig. tio-2 does not have peaks indicative of amorphous carbon. At relatively low reaction chamber pressures such as those indicated in column 2 of Table 1, the other reaction parameters such as laser power can be adjusted to yield pure samples of anatase titanium dioxide or rutile titanium dioxide.

Transmission electron microscopy (TEM) was used to determine particle sizes and morphology. TEM micrographs for the particles produced under the conditions in the four

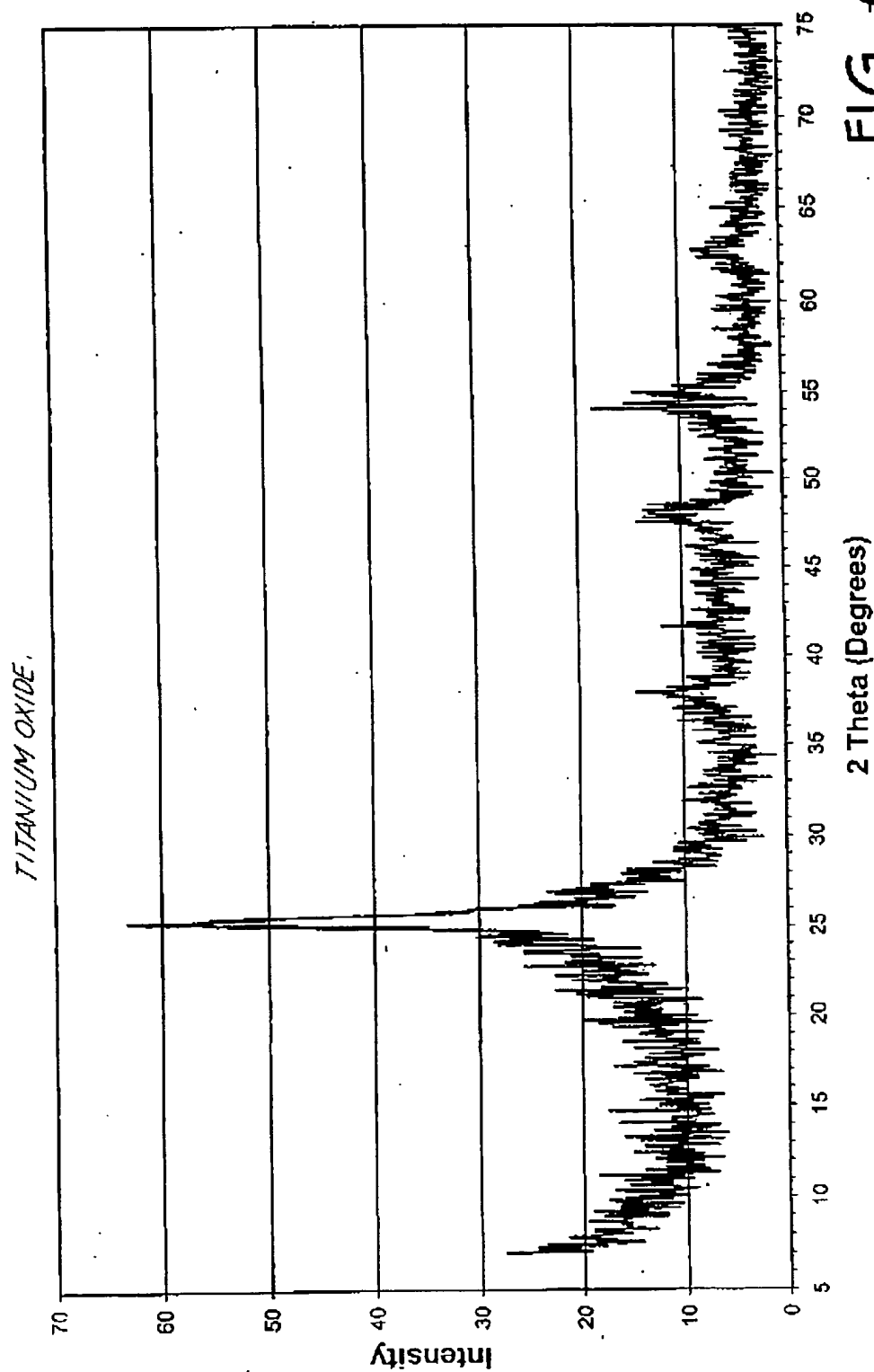
columns of Table 1 are displayed in Figs. tio-5 to tio-8, respectively. The particles in the TEM micrographs of Figs. tio-5 and tio-7 have visible coatings presumably corresponding to the amorphous carbon. The particles are quite spherical in shape. The cores are presumably formed from titanium oxide. Also, the cores seem to have facets corresponding to the crystal lattice of the titanium oxide.

An examination of a portion of the TEM micrograph in Fig. tio-7 yielded an average particle size of about 25 nm. The corresponding particle size distribution is shown in Fig. tio-9. The approximate size distribution was determined by manually measuring diameters of the particles distinctly visible in the micrograph of Fig. tio-7. Only those particles having clear particle boundaries were measured to avoid regions distorted or out of focus in the micrograph. Measurements so obtained should be more accurate and are not biased since a single view cannot show a clear perspective of all particles. It is significant that the particles span a rather narrow range of sizes.

The particle size distribution for the mixed phase materials produced under the conditions specified in column 2 of Table 1 is shown in Fig. tio-10. The distribution in Fig. tio-10 is broader than the distribution shown in Fig. tio-9. The distribution in Fig. tio-10 is consistent with a bimodal distribution with one component of the distribution corresponding to the anatase phase and a second component of the distribution corresponding to the rutile phase. This bimodal distribution suggests that the two phases are segregated into different nanoparticles. Nevertheless, the distribution in Fig. tio-10 is relatively narrow and does not have a tail, i.e., no particle have diameters significantly larger than a few multiples of the average diameter.

The particles had a dark color upon visual inspection. The darkness apparently was due at least partially to the deposits on the particles of amorphous carbon. The particles formed under the conditions indicated

in the second column of Table 1, which apparently lacked carbon deposits, were less dark than the other particles. Thus, there may have been a contribution to the darkness besides the presence of carbon deposits.



## Titanium Oxide

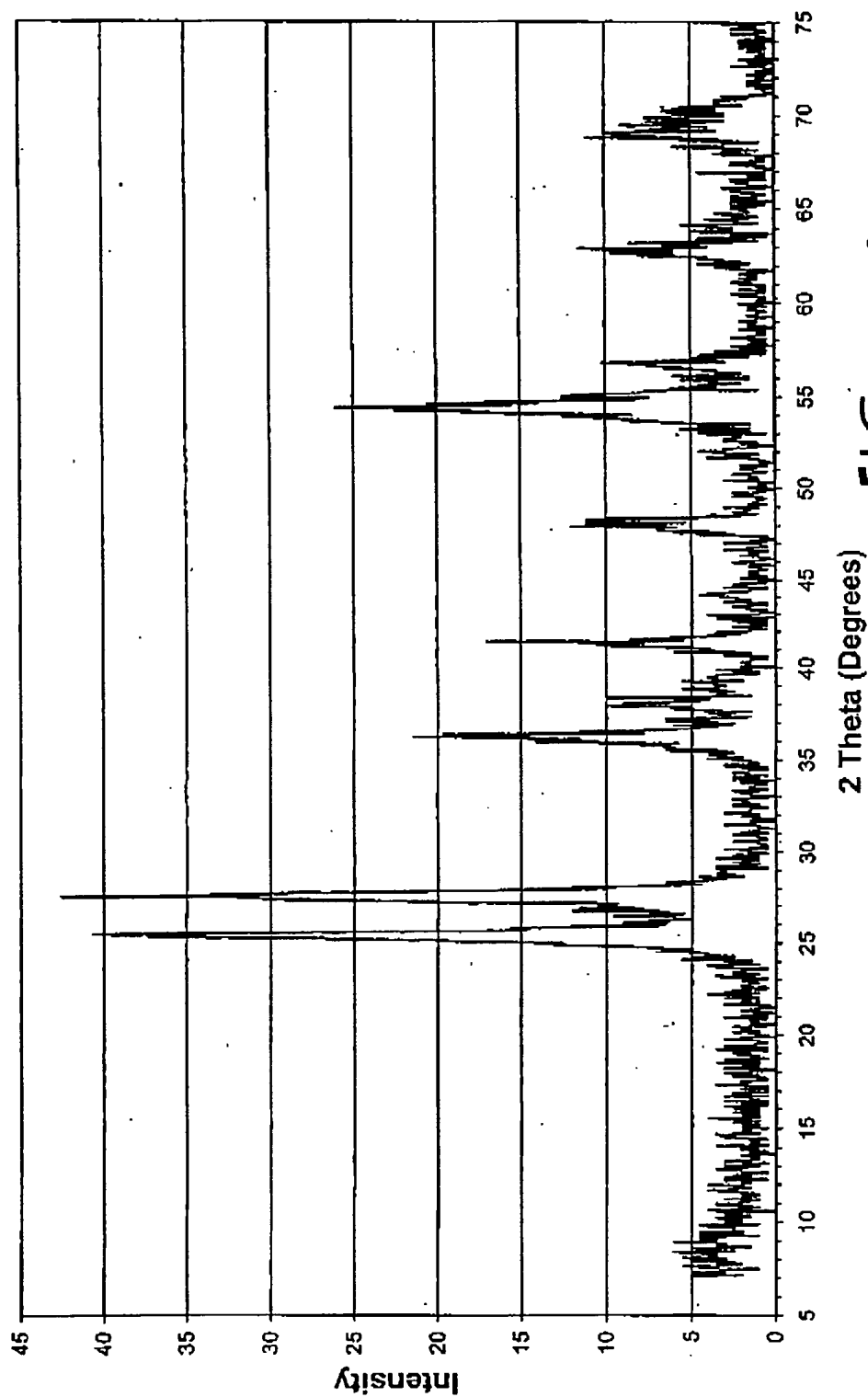
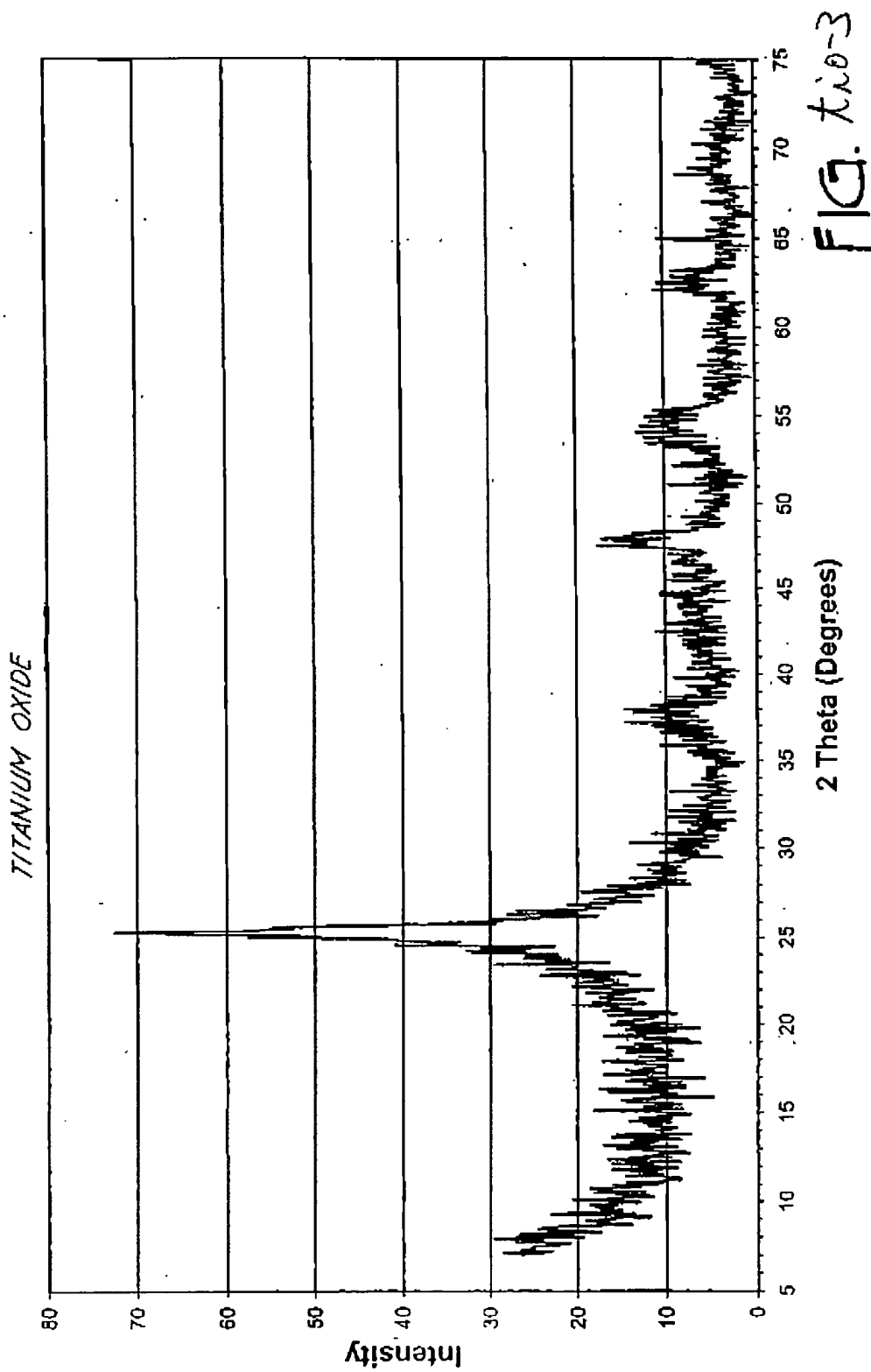


FIG. tio-2



## Titanium Dioxide

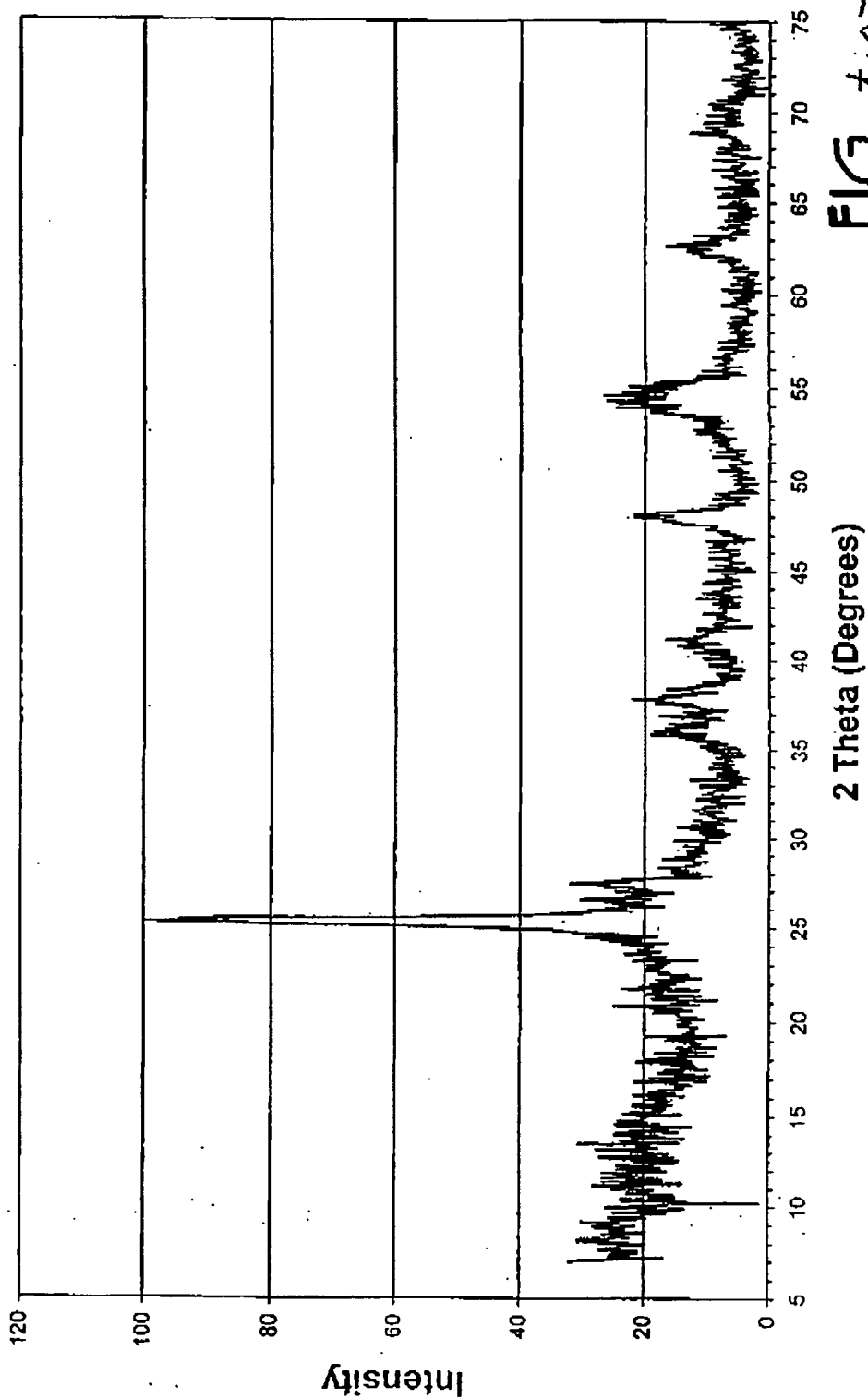


FIG. 10-4

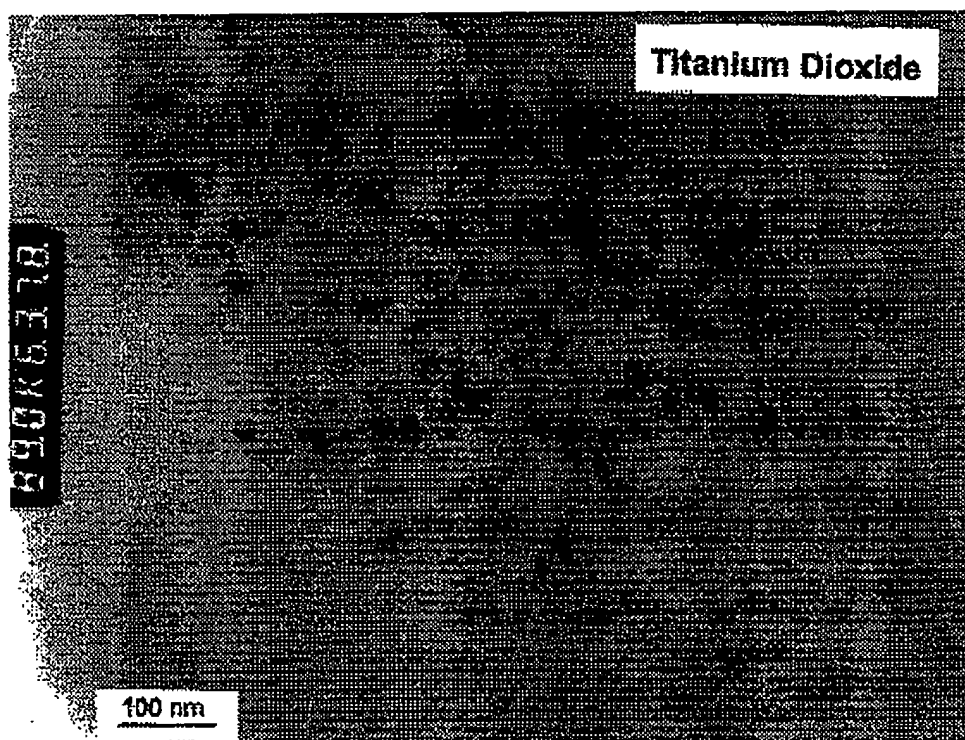


FIG. *tio-5*

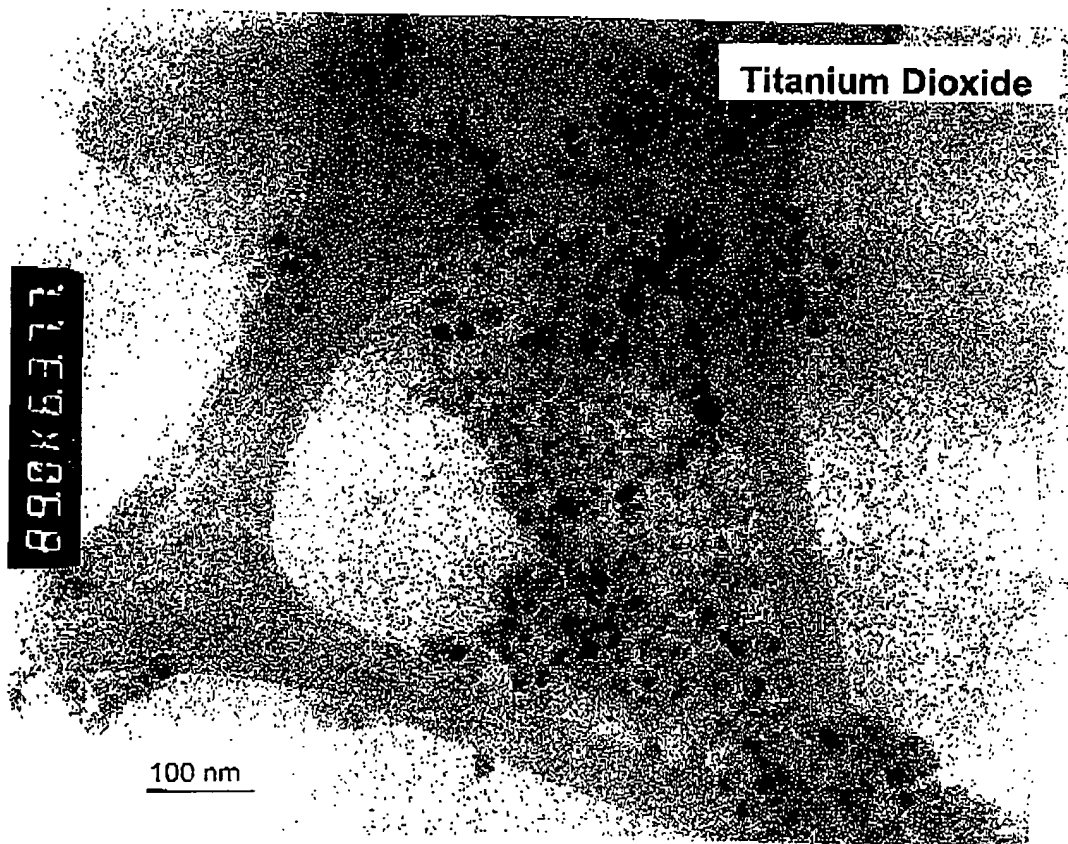


FIG. tio-6

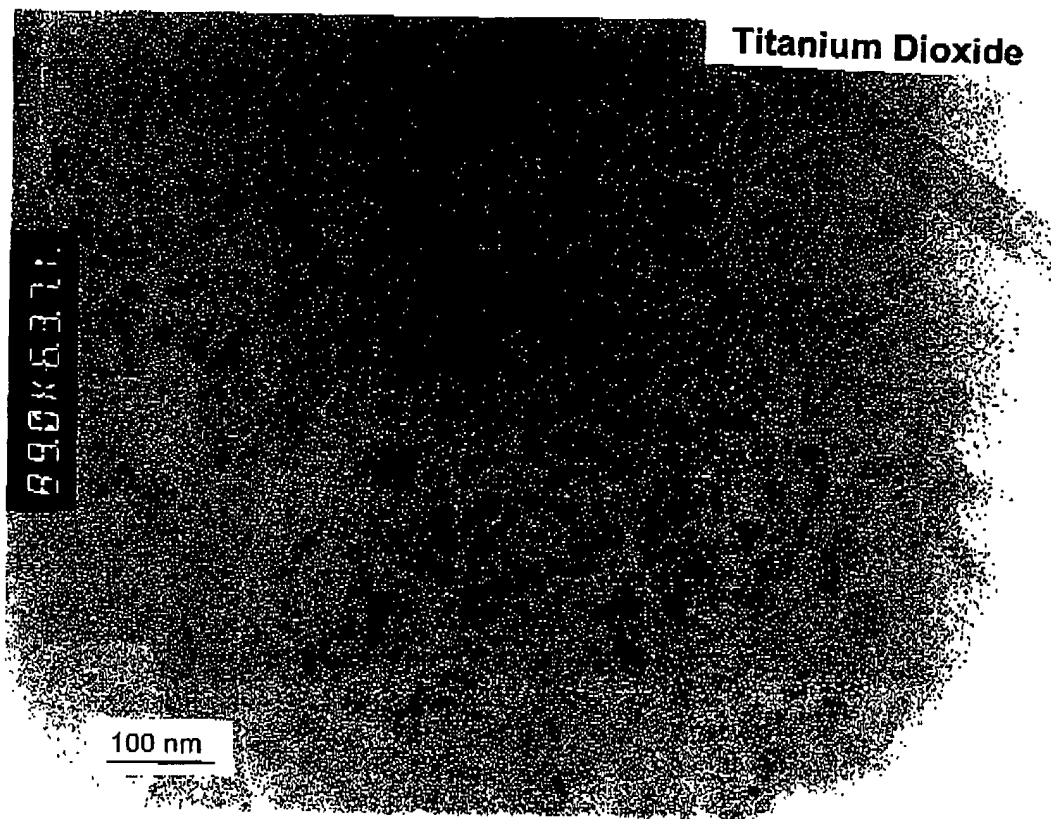


FIG.  $\text{tio-7}$

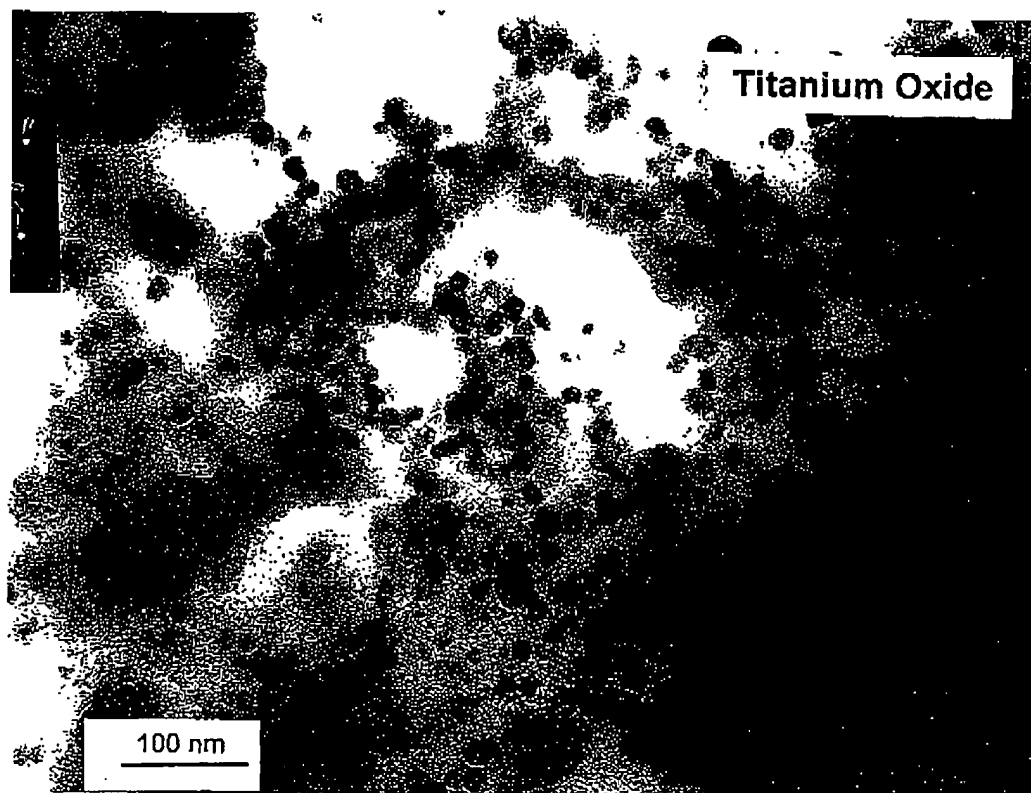


FIG. tio-8

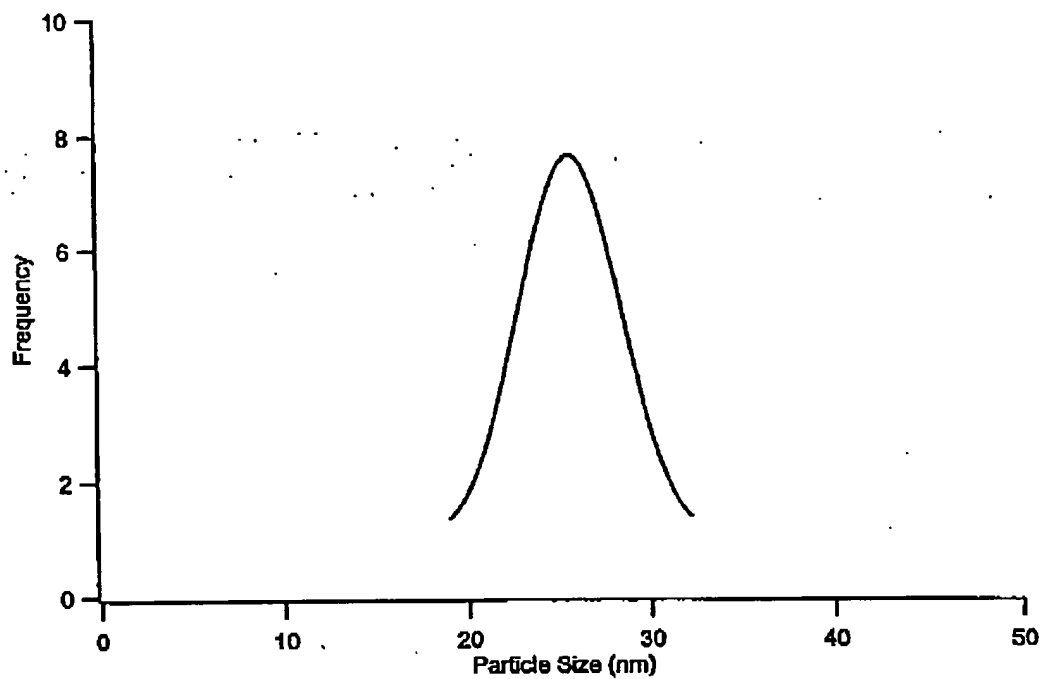


FIG. tio-9

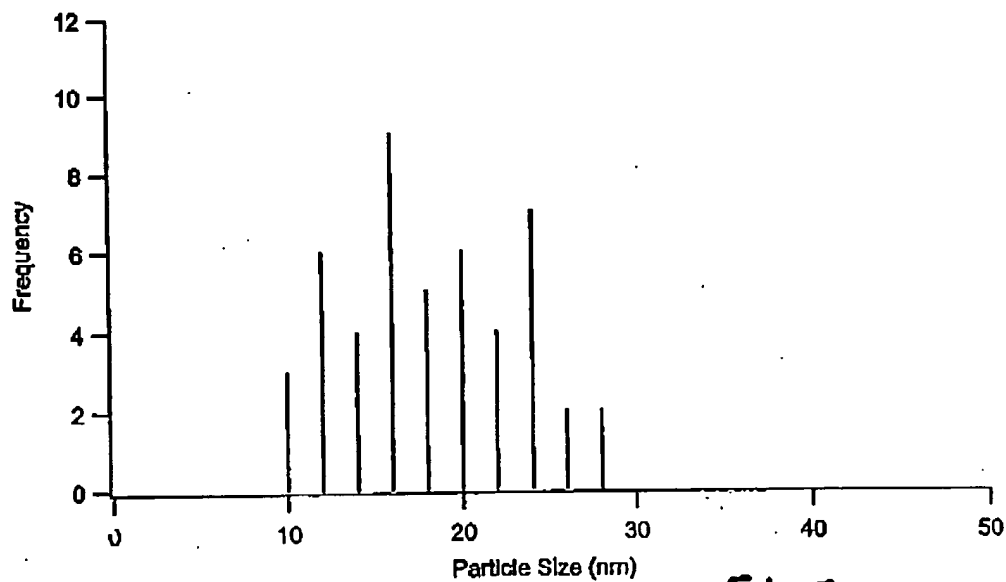


FIG. tio-10

From WO 02/058,928

Amended to refer directly to  
particle synthesis exampleExample - Absorption Spectrum From Titanium Oxide Particles

This example provides a description of the formation of well dispersed dilute solutions of titanium oxide nanoparticles produced by laser pyrolysis.

 from  
U.S.  
6,382,531

The suspensions were formed using each of the three types of  $\text{TiO}_2$  particles produced by laser pyrolysis. The three powders were separately suspended in water, ethanol, dimethyl sulfoxide (DMSO), cyclohexane, cyclohexanone and phentydrone (1,2,3,4-tetrahydro-9-fluorenone, THF). The suspensions were formed with 9.75 milligrams (mg) of  $\text{TiO}_2$  powders in 13 grams of liquid resulting in a suspension with 0.075 wt%  $\text{TiO}_2$ . The samples were sonicated for 2 hours each in a sonicate bath. Then, the relative sedimentation of all the samples was visually detected in parallel for two weeks.

The results are presented in Table 2. The relative sedimentation of all the samples is marked in parentheses following observations after two weeks and then several months, with number 1 being the worst and number 7 being the best.

TABLE 2

Solvent	$\text{TiO}_2$ -1	$\text{TiO}_2$ -2	$\text{TiO}_2$ -3
water	very poor (3) 100% settled	very poor (1) 100% settled	very poor (2) 100% settled
cyclohexanone	very good (4) ~90% suspended	very good (5) ~85% suspended	excellent (7) ~100% suspended
cyclohexane	very poor (2) 100% settled	very poor (2) 100% settled	very poor (3) 100% settled
ethanol	excellent (6) ~95% suspended	good (4) >75% settled	excellent (6) ~90% suspended
THF	excellent (5) ~95% suspended	excellent (7) >30% settled*	very poor (4) ~100% settled
DMSO	very good (7) ~80% suspended*	very good (6) >50% settled*	poor (5) >70% settled
toluene	very poor (1) 100% settled	very poor (3) 100% settled	very poor (1) 100% settled

\* The suspended particles remained suspended for months.

The best suspensions for a short term period (i.e., minimum sedimentation is observed after two weeks) were formed with cyclohexanone and ethanol. THF also suspended one of the samples extremely well. These suspensions exhibited no or only slight deposition of particles even after two weeks. The results suggest that solvents/dispersants with medium polarity provide the best suspensions, while solvents with very low or very high dielectric constant are not as suitable.

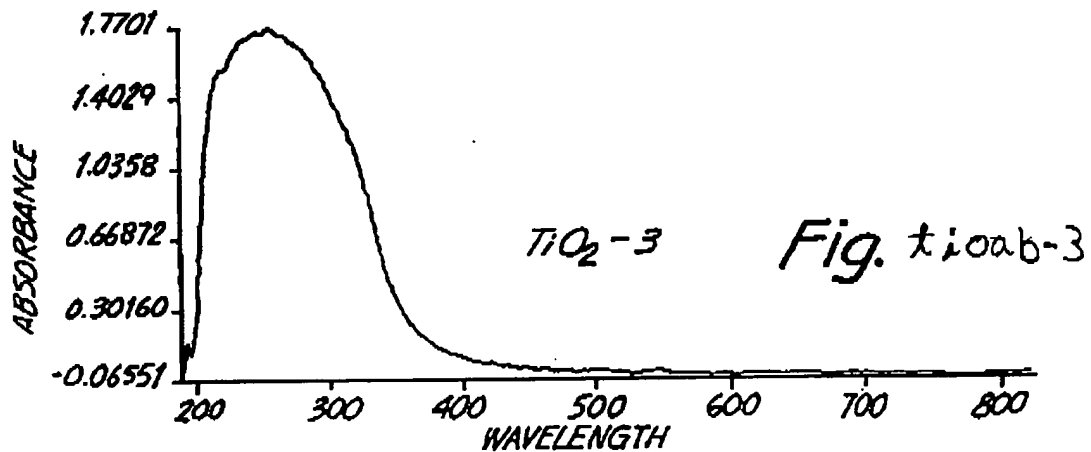
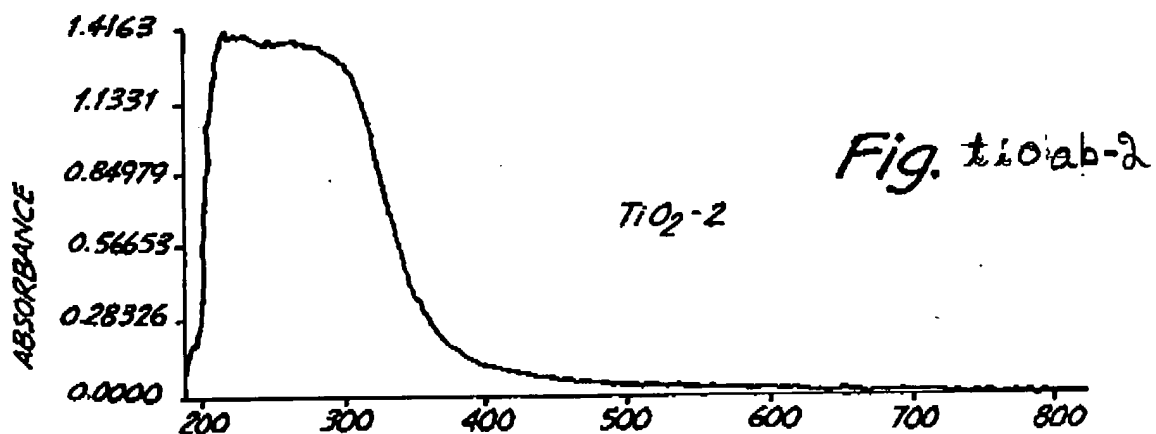
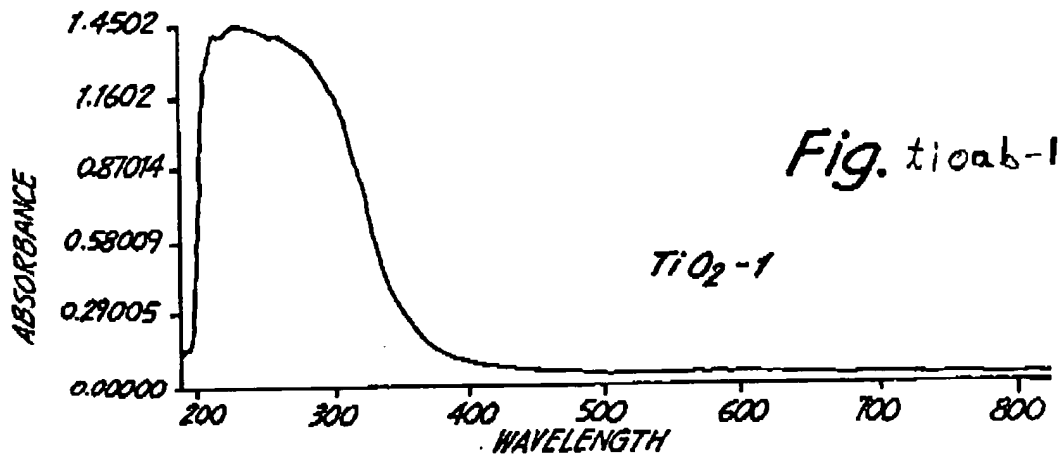
Secondary particle size in the suspensions were evaluated with a Horiba Particle Size Analyzer (Horiba, Kyoto, Japan). Analysis with the particle size analyzer showed good dispersion/low agglomeration with all dispersants that suspended well the particles. Generally, all of the suspended particles were in the size region below 80 nm, with broader distributions with lower average particle size.

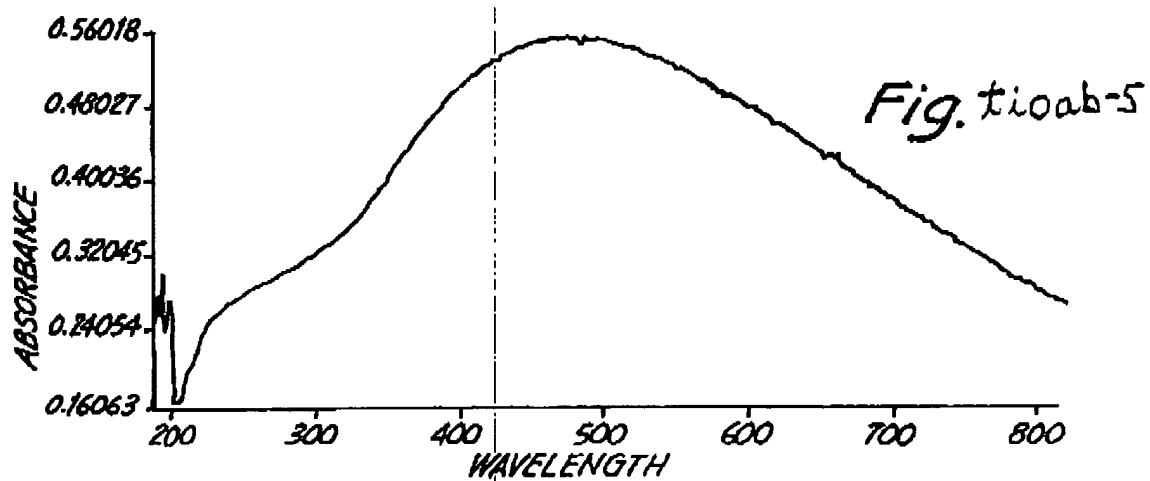
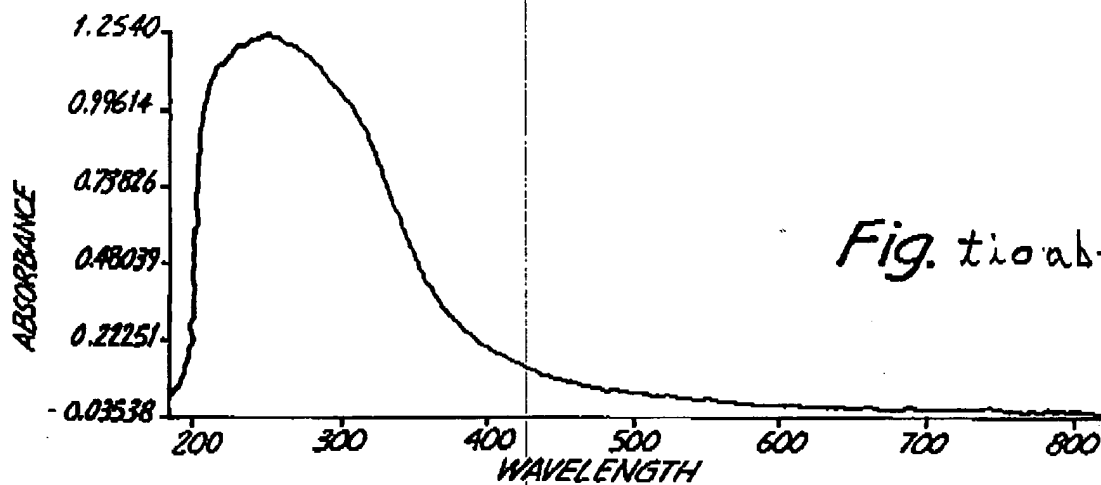
Since the particle size analyzer had a detection cut-off at 30 nm, an internal standard was used to estimate the number of particles with diameters less than 30 nm. A well characterized commercial  $\text{TiO}_2$  powder (R706 average particle size 0.36 microns with a coating of  $\text{Al}_2\text{O}_3$ , DuPont, Wilmington, DE) was mixed with the nanoparticles in a 1 to 1 by weight ratio. The resulting suspension was analyzed with the Horiba particle size analyzer. Less than about 10 percent of the nanoparticles were observed. Thus, most of the nanoparticles had a diameter less than about 30 nm and are undetected by the particle size analyzer. Nevertheless, the trends measured with the particle size analyzer were indicative of the level of agglomeration. In particular, good dispersions were formed with solvents that performed well in suspending the particles.

Absorption spectra were obtained for titanium oxide particles in ethanol at a concentration of 0.003 weight percent. The spectra for  $\text{TiO}_2$ -1,  $\text{TiO}_2$ -2 and  $\text{TiO}_2$ -3 samples are shown in Figs. tioab-1 to tioab-3, respectively. For

comparison, similar spectra were obtained for two commercial  $\text{TiO}_2$  powders dispersed in ethanol at a concentration of 0.0003 weight percent, which are shown in Figs. tioab-4 and tioab-5. The first commercial powder (Fig. tioab-4) was obtained from Alfa Aesar, Inc., Ward Hill, MA and had an average particle size of 0.17 microns. The second commercial powder (Fig. tioab-5) was obtained from Aldrich Chemical Company, Milwaukee, WI, and had an average particle size of 0.26 microns.

The absorption spectra of the  $\text{TiO}_2$  in Fig. tioab-5 is exemplary of bulk  $\text{TiO}_2$  with a large absorption in the visible and infrared portions of the spectra. In contrast, the absorption spectra of the powders in Figs. tioab-1 to tioab-4 have very reduced absorption in the visible and infrared portions of the spectra and enhanced absorption in the ultraviolet. This shift and narrowing of the absorption spectra is due to the reduced size of the particles. The spectra of the laser pyrolysis materials in Figs. tioab-1 to tioab-3 have an even more reduced visible absorption and a narrower and enhanced ultraviolet absorption relative to the powders yielding the spectrum in Fig. tioab-4.



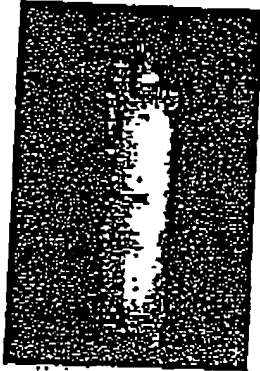


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## Planargard Disposable Filters



*Planargard Disposable filters offer convenience and versatility when filtering CMP slurries, with full compatibility for both oxide and metal slurries. The unique design, employing standard Flaretek fittings and a fully integrated polypropylene filter and shell, provides automatic draining capabilities, reduced holdup volume, and minimized exposure during filter change-out. Planargard Disposable filters avoid problems caused by non-standard fittings, and allow users to choose a range of options from maximum filter life to fewer wafer defects. Planargard Disposable filters — the result of Millipore's extensive applications expertise — represent the latest advances in proven filtration capabilities.*

### Specifications

#### Materials of Construction

All polypropylene construction with no O-rings or elastomeric seals.

#### Connections

##### Inlet/Outlet

1/2 in Flaretek fittings, 1 in Flaretek fittings

##### Vent/Drain

1/4 in Flaretek fittings

#### Operating Conditions

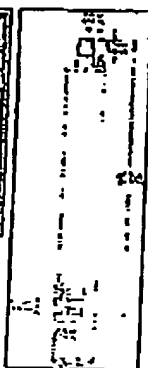
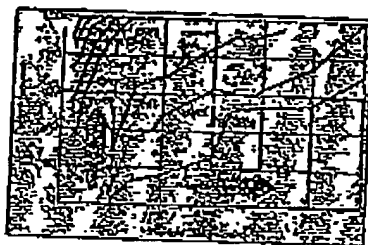
##### Maximum Pressure

4.1 bar (60 psi) at 25 °C

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Page 2 of 3

ORDERING INFORMATION									
C	M						0	6	
Filter Choice		Length		Package Quantity					
P3		1 = 10 in		06 = 6 per package					
P5		2 = 20 in							
P7									
P9									
11		1 = In-Line		1 = 1 in Flaretek					
13		U = U-Line		inlet/outlet fittings					
16				(in-line only)					
				5 = 1/2 in Flaretek					
				inlet/outlet fittings					
				(U-line only)					
Note: Filter choices CM13 and CM16 are not available in the U-line configuration.									



Planargard disposable In-Line filter with 1 in Flaretek inlet/outlet fittings and 1/4 in Flaretek vent/drain fittings

Planargard disposable U-Line filter with 1/2 in Flaretek inlet/outlet fittings and 1/4 in Flaretek drain fittings

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Table of Contents | Search

## Millipore Planargard Filters



### Description

- Graded-density filter removes agglomerated large particles before they can scratch wafer surfaces
- Removes disruptive particles without removing small working particles, ensuring consistent slurry delivery to your process
- All polypropylene construction provides excellent compatibility with high and low pH slurries

### Applications

- Point-of-use and bulk filtration of MLD slurries for chemical mechanical planarization (CMP)

### Specifications

#### Materials

All-polypropylene construction  
O-rings  
EPR

#### Cartridge Dimensions

Diameter  
70 mm (2.75 in)  
Length  
10": 264 mm (10.4 in)  
20": 512 mm (20.2 in)  
30": 761 mm (30 in)

#### Connections

Code 0 2-222 double O-ring

Munro product m

## Operating Conditions

Maximum Forward Differential Pressure

4.8 bar (70 psi) at 20 °C

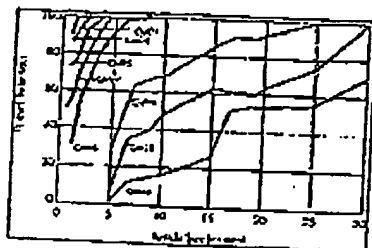
## Filter Selection Guide

Filtration Objective	Location	Suggested Product
Minimize wafer defects	Point of use	CMP1, CMP3, CMP5
Extend filter life	Point of use	CMP7, CMP9, CM11
Remove gross contaminants	Point of use	CM13
Reduce large particles formed in shipping container	Intake	CM14, CM16, CM18
Reduce large particles and gels formed during dilution	Post-dilution	CMP9, CM11, CM13
Continuous cleaning	Recirculation loop	CM14, CM16, CM18

**ORDERING INFORMATION**

C	M			O		E	O	6
Filter Choice				Length	Package Quantity			
P1 P3 P5 P7 P9 11 13 14 16 18				1 = 10" 2 = 20" 3 = 30"	06 = 6 per package			
Cartridge Code				O-Ring Material				
0 = Code 0				Delivered with EPR				
[2-222] O-Ring				O-Rings installed on cartridge				

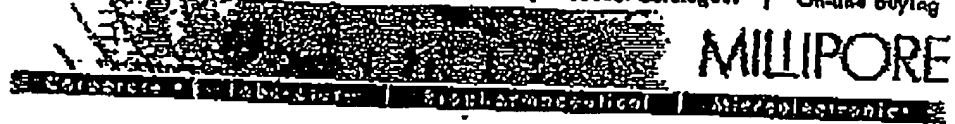
Note: All Planagard filters are delivered with EPR, Code 0 (2-222) O-Rings installed.  
All Planagard filters are shipped 6 per package.



Actual Particle Retention in Oxide SlurryXXX

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### Guide to Millipore Products for CMP Processes

The CMP slurry filtration challenge is to remove large particles and agglomerates from slurry that can cause defects, without changing slurry performance.

## Point-of-Use Filtration



CMP Slurries introduce millions of abrasive particles to the wafer, potentially causing scratches on the surface. Point-of-use filtration is the most effective location for reducing the number of defect-causing particles. We have seen up to a ten-fold reduction in light point defects by using Planargard filters at the point-of-use.

For more applications information, see our Millipore technical document, MA071 POU Filtration of Silica-Based CMP Slurries Using Planargard Filters

by Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos.

### Point of Use Filtration Selection Guide

Find the type of slurry used in your process and select the filter for specific product information.

Slurry Type

Recommended Filter

Fumed Silica Oxide  
(Diluted Cabot<sup>®</sup> SS25 and SC1,  
SS12, SC112; Rodel<sup>®</sup> ILD1300,  
ILD1200)

- Planargard Cartridge Filter,  
membrane type CMP5
- Planargard Disposable  
Filter, membrane type  
CMP5
- Planargard Cartridge Filter,  
membrane type CMP3
- Planargard Disposable  
Filter, membrane type  
CMP3

Colloidal Silica Oxide  
(Klebosol<sup>®</sup> slurries)

- Planargard Cartridge Filter,  
membrane type CMP5
- Planargard Disposable  
Filter, membrane type  
CMP5
- Planargard Cartridge Filter,  
membrane type CMP3
- Planargard Disposable  
Filter, membrane type  
CMP3

Silica-based Metal  
(Cabot SSW200)

- Planargard Cartridge Filter,  
membrane type CMP5
- Planargard Disposable  
Filter, membrane type  
CMP5

Metal (non-silica based)  
<2% solids  
>2% solids

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Application Note: MA071 POU Filtration of Sil

Page 3 of 12

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Millipore MicroElectronics DivisionLiquid Application Notes Database*Millipore MicroElectronics Division Technical Document MA071*

POU Filtration of Silica-Based CMP Slurries Using Planargard Filters

Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos

### Introduction

Chemical Mechanical Polishing (CMP) has become an enabling technology in semiconductor device manufacturing. The CMP process uses submicron (30 - 200 nm) silica slurries at a typical concentration of 10-30% solids. Typical silica slurries can contain a small number (104 to 106 counts/ml) of > 1.0 micron particles, which could potentially cause defects (microscratches) on the planarized wafer surfaces. The slurry solution presents unique challenges in delivery, filtration, and particle measurement.

The silica slurries used in the CMP process are stabilized suspensions of fine particles, typically 30 to 200 nm in size with concentrations ranging from 10 to 30%, in aqueous solutions with a specific pH. These slurries, which may need on-site dilution in the FABs, are applied directly onto the rotating pad to polish wafers and achieve global planarity. Fumed silica-based and "colloidal" silica-based slurries are the two families that are commercially available.

Fumed silica slurry is manufactured in two steps: 1) production of fumed silica (three-dimensional branched chain aggregates) by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame [2]; 2) dispersion of fumed silica in aqueous medium with certain additives. Commercial fumed silica CMP slurries normally contain silica aggregates with mean particle sizes ranging from 100 nm to 200 nm. Figure 1a shows a SEM picture of typical silica aggregates.

"Colloidal" silica is produced from a dilute aqueous solution of water glass through deionization/nucleation, polymerization, particle growth and concentration steps [3]. All process steps are in the liquid phase. The silica particles formed are normally spherical. Commercial colloidal silica CMP slurries have particles with mean sizes ranging from 30 nm to 50 nm. Figure 1b shows a SEM picture of typical colloidal silica particles.

### Filtration Needs

The typical specification for commercial silica slurries includes percent solids, pH, specific gravity, mean particle size and general (bulk) particle size distribution. However, a small number of "large" particles (>1µm) have been found which fall outside of the specified size distribution. These particles, which can be aggregates, agglomerates. SEM images shown in Figures 2A and 2B confirm their existence. These large particles may come from agglomeration or local drying of slurry on shipping containers and in the distribution system. Gels may form due to pH shocks during dilution or

## Application Note: MA071 POU Filtration of SiL

Page 4 of 12

temperature fluctuations during shipment and storage.

There is no definitive information available on what size or type of particles can cause microscratches and particle contamination on wafer surfaces. However, higher numbers of "large particles" have been found to cause higher incidence of microscratches and particle contamination on polished wafers. Slurry filtration has proven to be beneficial in reducing wafer defects and increasing yields in CMP processes[1].

A control oxide CMP polishing experiment was conducted using a commercial fumed silica slurry contaminated with 5 m silica particles (~ 104 particles/ml). The polishing were performed on an IPEC/Westech 472 tool using a standard oxide CMP recipe. The bare wafers were deposited with 1000 nm PECVD SiO<sub>2</sub> before polishing. Figure 3 and 4 show the surface scan results on the wafers polished with and without point-of-use filtration. A ten-fold reduction of light point defects was achieved by using a Planargard CMP5 filter at the POU.

### Slurry Characterization

The main challenge of slurry filtration is to selectively retain the small number of defect-causing "large" particles (i.e., 104 to 106 counts/ml greater than 1  $\mu$ m) without retaining the desirable, small particles (30 to 200 nm) present in very high concentration (> 1015 counts/ml). There should be no measurable changes to the slurry's percent solids concentration and bulk particle size distribution before and after filtration. Therefore, filters to be used in CMP slurry filtration should be evaluated in slurry to validate their performance for the following attributes:

- Retention efficiency for "large" particles
- % solids content and bulk particle size distribution before and after filtration
- Throughput (Lifetime)

#### 1. Detection of "Large" Particles

Quantitative determination of "large particles" is required to determine filter retention and a correlation between large particle concentration and wafer defects. There is no commercial particle counters available that can be used to detect the large particles (104 to 106 counts/ml >1  $\mu$ m) in the presence of bulk slurry particles (> 1015 counts/ml), without substantial sample dilution. Various particle counters were evaluated to determine their ability to detect the large particles with maximum tolerance to high concentrations of small particles (minimum sample dilution) and with ease of operation. A light scattering instrument was selected for the slurry application.

A typical schematic of the particle counting system, shown in Figure 5, includes continuous on-line dilution of slurry. The dilution factor should be high enough to minimize the interference caused by small particles in the slurry. Figure 6 shows a typical profile of large particle concentrations in oxide slurry before and after filtration. Filter retention for particles of a specific size can then be calculated based on the particle concentration before and after filtration.

#### 2. Measurement of Bulk Particle Size Distribution and Percent Solids

Bulk particle size distribution (PSD) can be measured by many techniques [4]. The two most commonly used techniques in CMP slurries are light scattering and chromatography (i.e., capillary hydrodynamic fractionation, or CHDF). The light scattering instrument used for slurry PSD measurement is based on photon correlation spectroscopy (PCS), also referred to as quasi-elastic light scattering (QELS) or time-dependent light-scattering. With PCS, the size information is obtained from the time dependent fluctuation of scattered intensity due to concentration fluctuations resulting from Brownian motion of particles[4].

CHDF is based on the size exclusion effects that occur when a dispersion of particles flows through a

Application Note: MA071 POU Filtration of Sil...

Page 5 of 12

capillary tube. Laminar flow in the capillary tube has a parabolic velocity profile. Smaller particles can reach the slower streamline close to the tube wall due to Brownian motion, while larger particles cannot. Therefore, large particles exit the capillary tube faster than smaller ones. CHDF can be used to measure particles between 15 nm and 1 micron with up to 1% solids [5]. This method was used for slurry particle size distribution measurement.

Another PSD instrument is based on acoustic attenuation spectroscopy. When acoustic waves propagate through a medium with suspended particles, the acoustic beam will be further attenuated by the particles by a variety of mechanisms. The acoustic attenuation spectrum can be detected and then inverted to obtain a mean particle size, a particle size distribution, and a dispersion concentration.

The percent solids in slurry can be calculated by drying a slurry sample of known weight.

#### Performance of Planargard™ Filters

Filter performance should be evaluated in slurry to determine: (a) retention efficiency for large particles; (b) percent solids content and bulk particle size distribution before and after filtration; (c) filtration throughput. This information is necessary for CMP process engineers to implement proper filtration.

Conventional microporous membrane filters will not work due to the high solids concentration in the slurry that forms a cake and plugs membrane quickly. Graded-density non-woven depth filters are preferred for this application. Filtration experiments were conducted with all-polypropylene graded-density Planargard™ filters using silica slurry in a single-pass configuration to simulate point-of-use applications. Feed and filtrate samples were taken and analyzed for large particle concentration, percent solids, and bulk particle size distribution. The filter retention efficiency is defined as:

Concentration in feed - Concentration in filtrate

$$\text{Retention} = \frac{\text{Concentration in feed} - \text{Concentration in filtrate}}{\text{Concentration in feed}} \times 100\%$$

The retention efficiency curves for various Planargard filters are shown in Figure 7.

Throughput was measured based on the differential pressure across the filter as a function of filtered volume. A typical plugging curve is illustrated in Figure 8, which shows a gradual increase in differential pressure across the filter. The differential pressure increases slowly initially, but then climbs rapidly as the filter reaches the end of life. Data analysis proves that the plugging process follows the complete plugging mechanism, which can be represented by a linear relationship between the inverse differential pressure across the filter (or  $DP_{min}/DP$  in dimensionless form) and filtration volume. In this mechanism, the pressure drop across the filter increases slowly at the beginning, which will then increase exponentially. The importance of understanding the plugging process is to determine the filter change out time before the pressure drop reaches the region of exponential increase.

To maintain process control the filter should not affect the slurry's composition and the filter should have consistent retention throughout its useful lifetime. Figure 9 shows that filter retention remains fairly constant throughout its lifetime. Table I shows that the filter does not change the percent solids and mean particle size in the slurry, which is extremely important for a CMP process. As long as the solids concentration is not changed before and after filtration, the filter will not alter the bulk particle size distribution. Figure 10 shows the slurry's bulk particle size distribution in the feed and filtrates at 60% and 95% of the CMP5 filter's throughput.

#### Implementation Strategy

## Application Note: MA071 POU Filtration of Sil...

Page 6 of 12

Implementation of silica slurry filtration depends on CMP process requirements, space availability, and the characteristics of the slurries. A number of field tests have demonstrated that point-of-use (POU) filtration at the tool can provide the most benefit in wafer defect reduction. Table II lists the normalized test results for oxide CMP with POU filtration.

Other filtration locations are at post-dilution, in the distribution loop, and at the slurry supply drum/tote.

It is strongly recommended that the implementation should start at POU filtration with higher retention filters to realize the maximum defect reduction benefits during CMP process development and qualification. To optimize the process, filtration at other locations can be used to supplement POU filter(s) and potentially to extend the life of POU filters.

### Conclusion

"Large particles" have been detected in CMP slurries using an optical particle counter. SEM evaluation of slurry particles on membrane filters confirmed their existence. These defect-causing large particles may come from agglomeration, local drying of slurry on shipping containers and in the distribution system, and gel formation due to pH shocks during dilution and temperature fluctuations.

Graded-density depth filter can be used effectively to remove the defect-causing large particles without measurable change to slurry composition. Field test data have demonstrated the benefits of filtration on wafer defect reduction during CMP processes.

The optimal strategy to implement filtration in CMP processes can be dependent on site, process, and slurry type. POU filtration with higher retention filters is recommended to realize maximum defect reduction benefits during CMP process development and qualification.

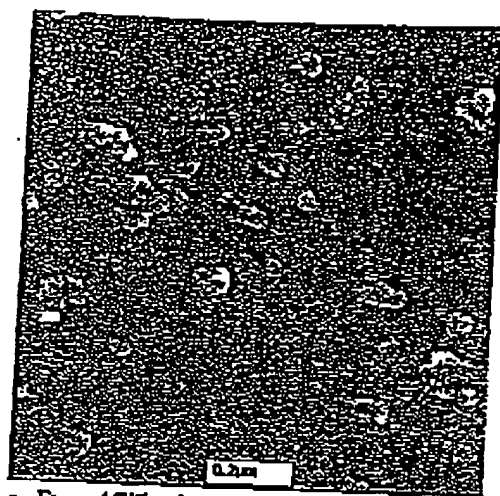
### References

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4. Barth, H.G and S.T. Sun, "Particle Size Analysis", Anal. Chem., 57, 151R-175R, 1985
5. J.G. DosRamos and C.A. Silcbi, "Size Analysis of simple and complex mixtures of colloids in the submicrometer ranges using capillary hydrodynamic fractionation", Chapter 19, ACS Symposium Series 472, 1990
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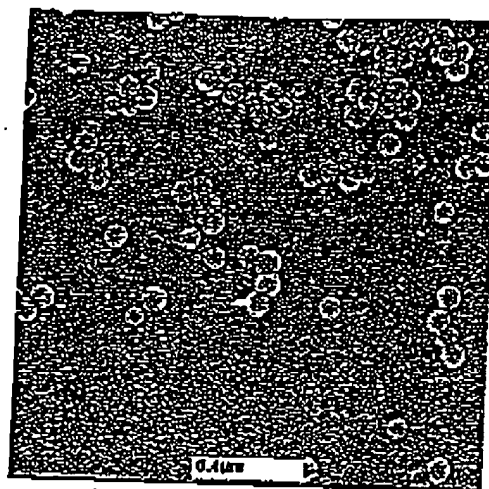
Figure 1  
Fumed Silica Aggregates and Colloidal Silica Particles

Application Note: MA071 P00 Filtration of Sil...

Page 7 of 12



a. Fumed Silica SEM



b. Colloidal Silica SEM

Figure 2  
"Large Particles" and Gel in Silica Slurries

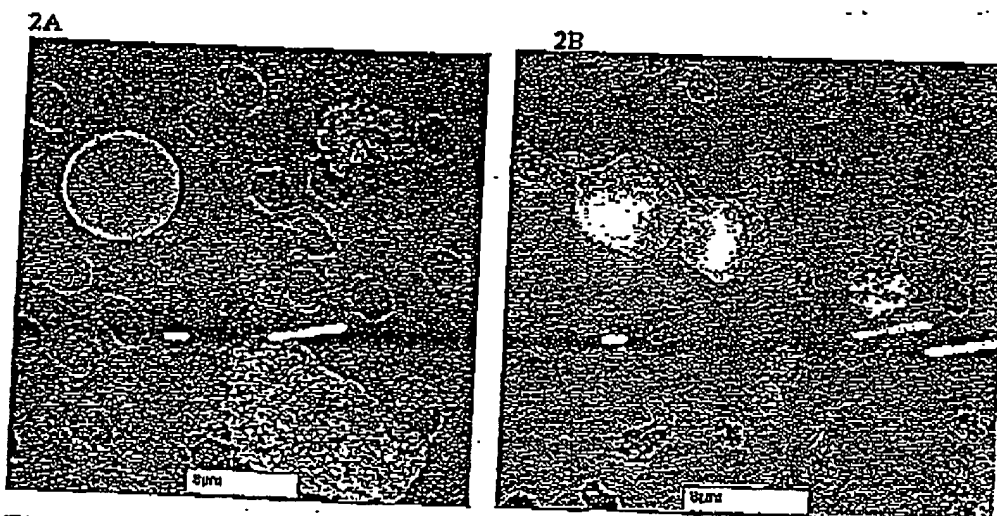


Figure 3 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with Filtered Slurry

Application Note: MA071 PCD Filtration of SiL

Page 8 of 12

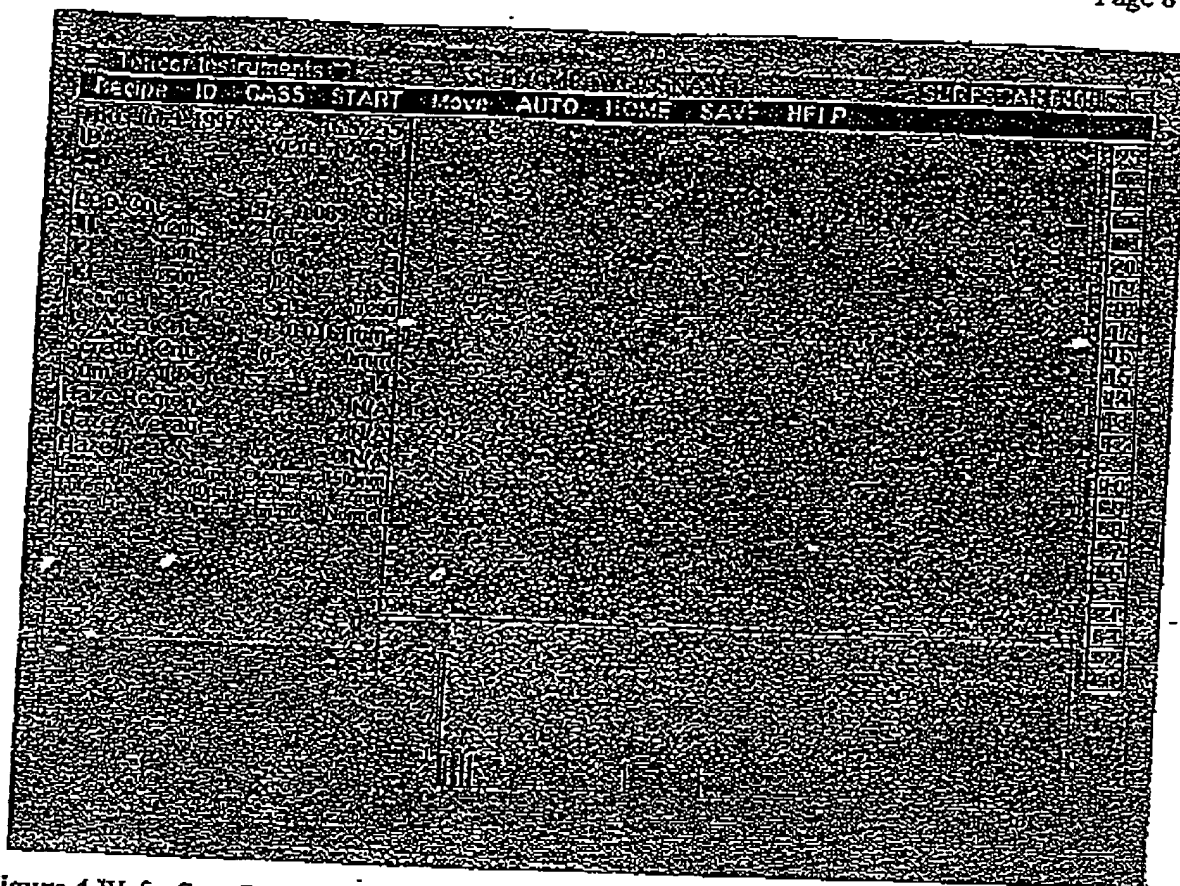
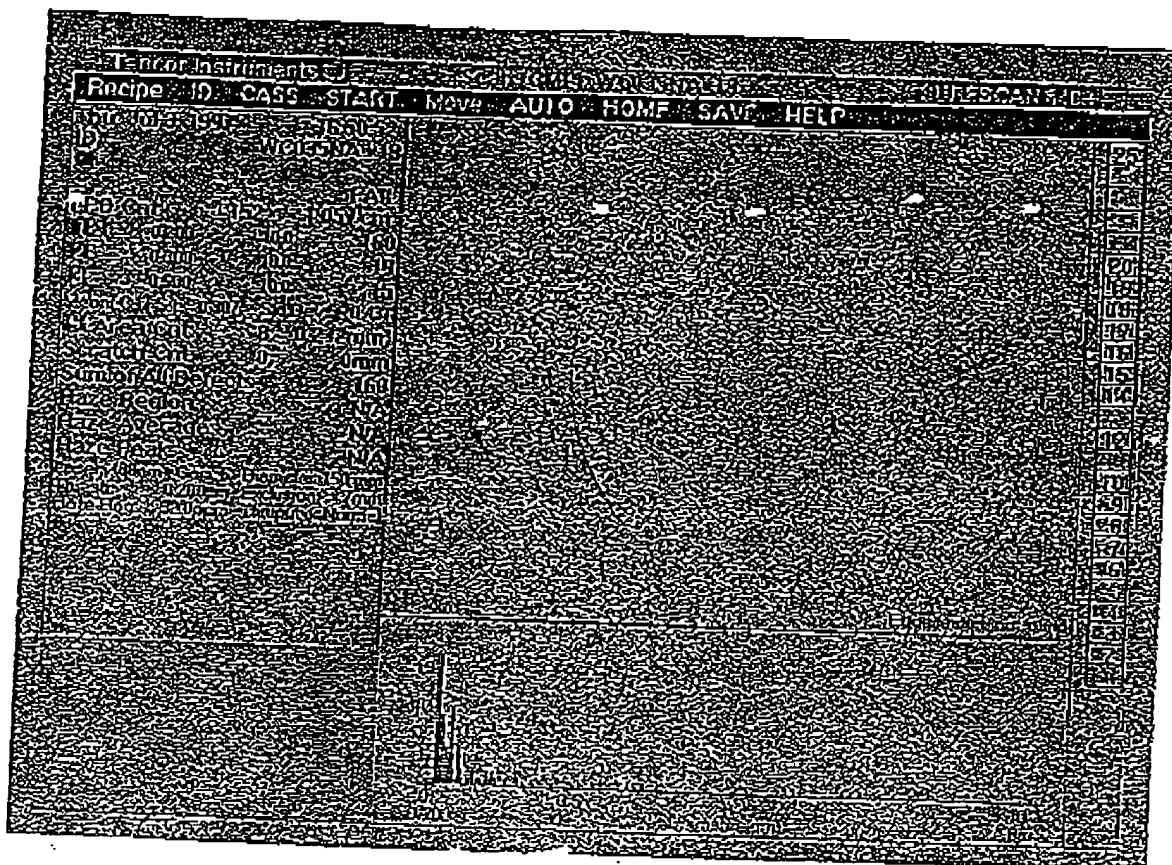


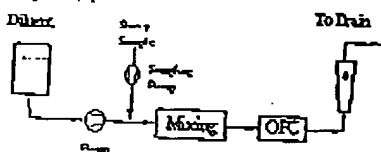
Figure 4 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with UN-filtered Slurry



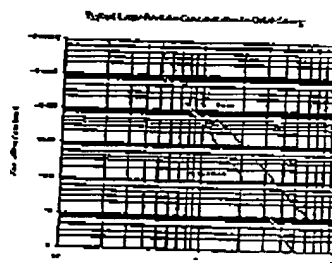
**Figure 5**

**Figure 6**

### Schematic of Large Particle Coupling System



**Figure 7**  
**Retention Efficiency of POU**  
**Planargard™ Filters**



**Figure 8**  
**CMP5 Filter Plugging Curve**  
**in Typical Silica Slurry**

## Application Note: MA071 POU Filtration of Sil...

Page 10 of 12

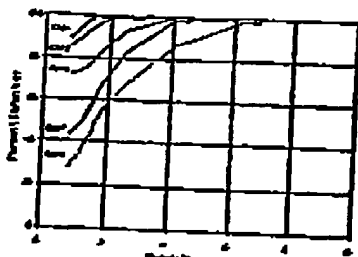


Figure 9  
Retention Efficiency and Filter Life

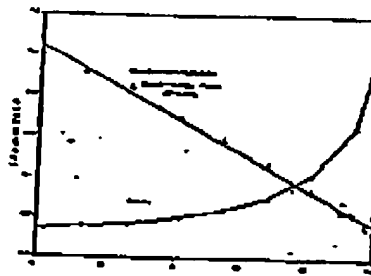


Figure 10  
Effect on CMP5 Filter on Slurry Bulk Particle Size Distribution



Table I  
Effect of Filter Plugging on  
Percent Solids and Mean Particle Size in Slurry

	Feed	60% Plugged	95% Plugged
%Solids	12.6	12.4	12.3
DW (nm)	192.3	197.	197.1
DN (nm)	93.3	93.3	94.1

DW: Mean diameter by weight  
DN: Mean diameter by number

Table II Field Test Results  
Light Point Defect (LPD) Reduction by POU Slurry Filtration

Customer	Slurry	Filters Tested at POU	Normalized LPD Levels
A	Fumed Silica	None	100
		CMP+CMP3	10
B	Colloidal Silica	None	100
		CMP7+CMP5	30
		CMP3+CMP1	9
C	Colloidal Silica	None	100
		CMP3	33

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Application Note: MA071 PCC Filtration of Sil.

Page 11 of 12

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